An Integrated Look at Insensitive High Explosives - Why Are They Insensitive?

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An Integrated Look at Insensitive High Explosives – Why Are They Insensitive?

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Abstract. The sensitivity of explosives to thermal or mechanical stimuli is controlled by the chemical and physical properties of the explosive materials. Integration of years of study allows identification and comparison of several of these properties for explosives with a range of sensitivity – HMX, Explosive D, LLM-105 and TATB. I identify key material properties that determine overall sensitivity, including the extremely safe behavior of Insensitive High Explosives, and discuss an approach to predicting the sensitivity or insensitivity of an explosive.

Introduction

The wide range of CHNO explosives exhibits an equally wide range of sensitivities to different stimuli, deliberate or accidental. Responses may range from violent detonations to very slow combustion (like wood) for identical stimuli, depending on the sensitivity of the explosive. The type of explosive known as Insensitive High Explosive (IHE) exhibits particularly benign behavior,[1] while other explosives show a wide range of responses.

In this paper I will identify and discuss many properties of explosives that, when integrated, form the basis for the insensitivity of some explosives. The primary focus is on properties of the molecular explosive. The effect of formulation variations will be of less emphasis, because the safety of today's IHEs results from the molecular properties, and also because formulation effects is a complex topic beyond the scope of this work. Comparison of properties for a range of explosives allows establishing the importance of each, and provides insight into development of new explosive molecules.

In considering explosive response to stimuli, the underlying mechanisms that together determine the response must be considered. Figure 1 shows an integrated view of many mechanistic steps that lead to explosive ignition and reaction under non-shock conditions.[2] A further description is available in an earlier publication.[3] The concept of hot spots is central to explosive initiation (shock and non-shock),[4] and represents the processes involved in the Ignition step in Figure 1. A simplified view of hot spot evolution is shown in Figure 2. The mechanisms in these two figures form the basis for the identification of material properties relevant to explosive sensitivity and insensitivity.
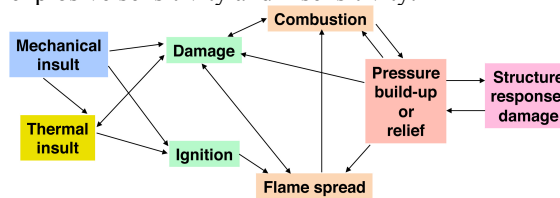

Fig. 1. Integrated view of physical and chemical processes that determine the behavior of explosives under mechanical and thermal stimuli.
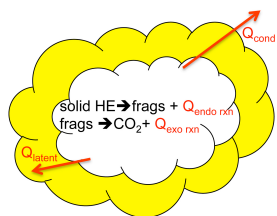
Fig. 2. Simplified mechanism of hotspot reaction, with several relevant properties.

I will consider these mechanisms and their underlying chemical and physical properties in the context of four explosives. Compared to the HMX baseline, Explosive D is somewhat less sensitive to shock and heat, and its properties form the threshold for IHE qualification in some tests.[1] TATB is the one explosive now qualified as an IHE[1] and is quite different from the others in sensitivity. LLM-105 is in between Explosive D and TATB in sensitivity, and is currently an IHE candidate.

Key Material Properties

The mechanisms in Figures 1 and 2 guide identification of key material properties that underlie explosive sensitivity. Of particular importance is that the generation of localized high temperatures is required for ignition of hot spots. Therefore material properties that lead to lower temperatures for a given stimulus and material properties that lead to a higher ignition temperature will have a major effect on sensitivity.

Molecular and Crystal Structure

Features in the molecular and crystal structure govern the inherent thermal stability of the molecule as well as influence the generation of high temperatures leading to hot spots. These structures are shown in Figure 3 for the four explosives under consideration.

Chemical structure factors that lead to highly stable molecules include aromatic rings, intermolecular hydrogen bonding, high symmetry, inclusion of amino and nitro groups (having both offers hydrogen bonding), presence of the strong $C-NO_2$ bond, and lack of the weaker $N-NO_2$ bond.[5] Inspection of the molecular structures in Figure 3 shows how these correlate with molecular sensitivity. HMX is not aromatic, has no amino groups, and contains $N-NO_2$ bonds. Explosive D is aromatic, LLM-105 is aromatic with amino groups, and TATB has all of the desired attributes.

The crystal structure of TATB is unique in being planar. This has at least two stabilizing effects – the close intermolecular spacing allows very strong hydrogen bonding, and physical deformation of the crystal can occur along the planar interfaces with very little disruption of the structure and therefore very little heating. This graphitic nature of TATB is a key to its inherent insensitivity. The structure of LLM-105, appears somewhat more planar than that of HMX or Explosive D, although I am unaware of shear strength data that would quantify this.
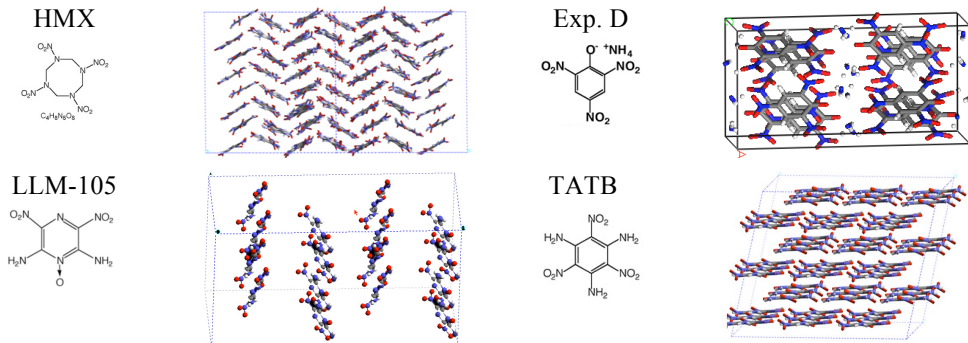

Fig. 3. Molecular and crystal structure of explosives under review.[6,7]

Thermophysical Properties

Properties that govern the build-up or dissipation of heat from ignition sites within an explosive include thermal conductivity and specific heat. Comparison of these properties across the four explosives is shown in Figure 4 and Table 1 (along with other properties discussed later). Here the high thermal conductivity of TATB promoted by its graphitic structure stands out. Explosive D is lowest in thermal conductivity, while the specific heat is about the same for all.
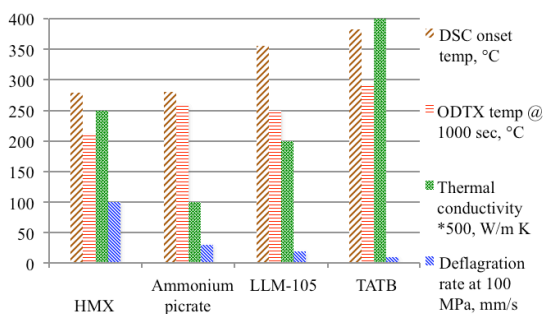

Fig. 4. Comparison of thermo-physical and chemical properties (note thermal conductivity values have been multiplied by 500 to fit to scale)

Table 1. Comparison of material properties; those leading to insensitivity are highlighted and in bold

Property	HMX	Exp. D	LLM-105	TATB
Aromatic	N	**Y**	**Y**	**Y**
Graphitic	N	N	N	**Y**
C_p, J/g K	1.0	1.1	0.9	1.0
Ignit. P-dep (DSC)	pos	**neg**	**neg**	pos
Deflag. rate P exponent	1++	0.8++	**0.8**	**0.9**

Thermochemical Properties

The thermal stability of the molecular explosive is the most important determinant of its insensitivity. This is commonly quantified by differential scanning calorimetry (DSC), which indicates the temperature to which an explosive must be heated before undergoing exothermic reaction. This onset temperature is shown in Figure 4 for each explosive, and we see that HMX and Explosive D are similar and much less thermally stable than LLM-105 and TATB.

DSC is most commonly measured at atmospheric pressure, but many stimuli that generate heat in explosive are mechanically driven and therefore occur at elevated pressure. For this reason, work is underway now to obtain DSC data on explosives at pressures up to ~ 5 MPa, and preliminary data are reported here in Figure 5.[8] These data were recorded at a very slow thermal ramp rate, 1°C/minute, to enhance the difference at different pressures, whereas typical DSC data are recorded at 5-10°C/minute.

Figure 5 shows that for HMX and TATB, the onset temperature decreases slightly as pressure increases. In contrast, the onset temperature increases with pressure for Explosive D and LLM-105. This is further illustrated in Figure 6, showing the shift in onset temperature above atmospheric pressure. The magnitudes of the shifts are approximately +0.4 K/MPa for Explosive D and LLM-105 and – 0.4 K/MPa for HMX and TATB.

To assess the importance of these data, consider a mechanical impact where the pressure is 50 MPa (1/2 kbar). The onset temperature for Explosive D and for LLM-105 increases by 20 K, with a corresponding reduction of 20 K for the onset temperature of HMX and TATB. The stability of Explosive D is improved over that of
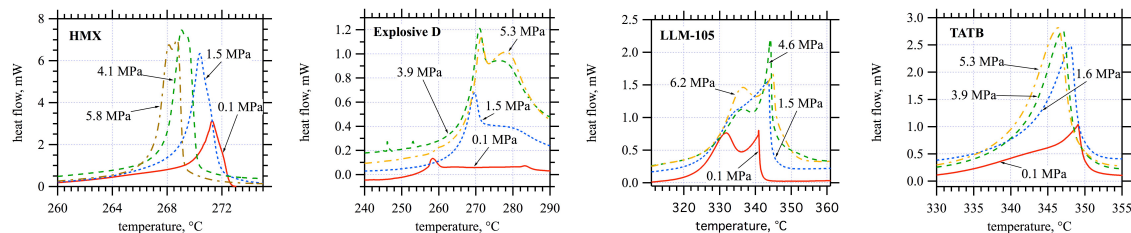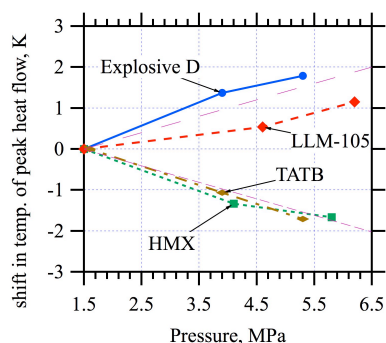

Fig. 5. High-pressure differential scanning calorimetry for four explosives. Ramp rate = 1K/min, sample size ~ 350 micrograms. Pinhole in pan to allow pressure equilibration in cell.

Fig. 6. Shift in peak exotherm temperature with pressure for four explosives. Unlabeled lines are for reference, showing shifts of ± 0.4 K/MPa.

HMX, consistent with their impact and shock sensitivities discussed below. LLM-105 is made more stable by this behavior, and TATB less stable, although for both of these materials the effect is perhaps less significant given their very high onset temperatures at atmospheric pressure.

Resulting Material Behaviors

The previous section primarily addresses molecular properties in the context of onset of ignition from localized heating, as shown in Figures 1 and 2. For example, DSC data indicate the onset temperature for a very small sample (< 1 milligram) with little influence from self-heating; this represents the initial reactions in hot spot formation and development. For subsequent reaction propagation and spreading to yield a response of the explosive, further material behaviors are also relevant. These result from a combination of several properties, but nonetheless give insight into the overall explosive stability. Such behaviors include ignition in the One Dimensional Time to eXplosion (ODTX) test, and the reaction propagation in the High Pressure Strand Burner (HPSB) test, as well as the change in material from physical or thermal damage.

One Dimensional Time to Explosion (ODTX)

In the ODTX, a 12.7 m diameter sphere of explosive is inserted into a preheated sample holder that confines the gases and provides a constant temperature boundary condition with essentially no initial void volume. The ODTX data of interest here are the time to explosion (when the 150 MPa confinement is broken) as a function of the initial temperature. Further details are given by Hsu.[9] The time to explosion includes the processes in Figure 2 – initial heat flow into the sample, heat generation in the sample, heat flow out of the hot spot – and thus represents an integration of several material properties. For example, self-heating of the ODTX sample is important, much more so than in the much smaller DSC sample, and is strongly affected by the thermal conductivity of the sample. ODTX data have been successfully used to develop global kinetic models for explosives.

ODTX data are shown in Figure 7 for the four explosives of interest. TATB shows the longest time to explosion, and HMX the shortest. Explosive D shows longer times to explosion than LLM-105 at low temperatures, with similar times at high temperatures. This is consistent with the lower thermal conductivity of Explosive D in Figure 4 – it simply takes longer for heat to penetrate into the ODTX sample by thermal conduction.
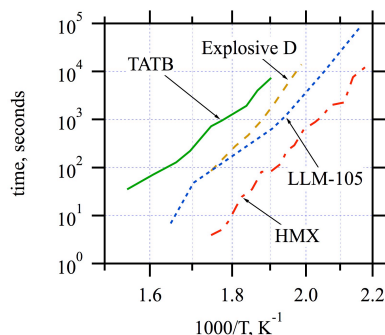

Fig. 7. One dimensional time to explosion (ODTX) data for the explosives of interest.

ODTX data have been measured with the apparatus vented so there is no pressure buildup. In addition, the effect of increased void volume has been reported for HMX.

For HMX (actually LX-04, 85 wt% HMX and 15 wt% Viton A), reducing the confinement pressure from 150 MPa to 50 MPa increased the initial void volume, and doubled the time to

explosion at 483 K from 110 to 210 minutes. In this case the increased initial void volume allowed decomposition gases to collect at lower pressures, and therefore allowed the decomposition to proceed for a longer time before ignition. Venting with no pressure buildup extended the reaction time to greater than 360 minutes.[10]

For TATB, data are available at 150 MPa and for vented samples. For pure TATB at 98% of theoretical maximum density (TMD), the results are unaffected by venting at temperatures > ~ 540 K (reaction times 0.5-17 minutes), while at 520 K the time to explosion is increased from 57 to 240 minutes when the sample is vented. For PBX 9502 (95 wt% TATB, 5 wt% Kel-F 800) at 98% TMD, there is essentially no change in time to explosion for confined and vented samples for all temperatures (reaction times 1-180 minutes).[11]

The ODTX results in Figure 7 indicate overall thermal stability, and represent an integration of many material properties. As such, the effects of these properties must be carefully considered. As noted above, the longer reaction times for Explosive D than LLM-105 despite lower DSC onset temperatures are consistent with the lower thermal conductivity of Explosive D. However, for samples heated internally rather than externally, as occurs in hot-spot formation, the lower thermal conductivity of Explosive D would reduce the heat loss from the hot spot and would increase its reactivity. This is consistent with the higher sensitivity of Explosive D as described below.

The effect of void volume (and hence buildup of gas pressure) or venting is complex and requires consideration of chemical mechanisms to unravel – this is beyond the scope of this work, and further discussion was provided by Burnham.[12,13] One might expect that vented samples would always show a longer reaction time than unvented samples, inasmuch as the released gases carry away energy that would otherwise contribute to the eventual runaway reaction – however, as described above, this is not seen with PBX 9502. This behavior shows the complexity of the integrated behavior in the ODTX test.

High Pressure Strand Burner

The propagation of reaction from an initial ignition site or hot spot occurs through deflagration of the explosive material. The deflagration behavior of explosives has been quantified using a high pressure strand burner, as previously described.[14,15] In brief, an explosive sample of 6.4 mm diameter and 64 mm length is ignited at one end and the progress of the deflagration along its length is monitored by time of arrival wires and pressure. Initial pressures may range from 5-400 MPa. The pressure vessel volume is sized so the pressure increases by roughly a factor of four during an experiment, providing the pressure dependence of deflagration across a range of pressures with only a few tests.

While deflagration rate data have been published for HMX, LLM-105 and TATB, the data for Explosive D were measured but not previously published; data are shown in Figure 8. Two features stand out. First, the deflagration rate at pressures below ~ 150 MPa lies below that of the LX-04 baseline (LX-04, with 15 wt% Viton A and 85 wt% HMX, shows uniform deflagration behavior across the entire pressure range and serves as a useful reference for comparison). Second, above ~ 150 MPa the deflagration rate shows the significant increases with somewhat erratic behavior that typifies deconsolidative burning.[14,15] In this regime, the high pressure is causing damage resulting in fracture and increased surface area before the deflagration front arrives in the material. The deflagration front spreads rapidly igniting the surface of the fractured particles, which subsequently burn at the inherent burn rate of solid intact explosive. This behavior corresponds to the damage, flame spread, and
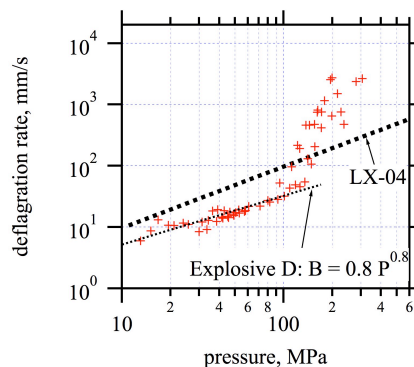

Fig. 8. Deflagration rate data for Explosive D. LX-04 (85 wt% HMX, 15 wt% Viton A) is shown for comparison.

combustion steps in Figure 1. Deconsolidative burning has been seen in HMX formulations with 5-10 wt% binder, while formulations with 15-20 wt% binder do not show this behavior.[14,15] Explosive D is a pure explosive without binder, and the onset of deconsolidative burning indicates that its mechanical properties such as strength and friability are more similar to low-binder than high-binder HMX explosives. The onset of deconsolidative burning has been shown to correspond to increased violence of thermal explosions.[16,17]

Deflagration data for HMX,[14,15] LLM-105 and TATB,[18] and Explosive D (Figure 8) are shown together in Figure 9. The straight line for HMX represents the behavior of high-binder HMX explosive (LX-04, 15 wt% Viton A) which does not exhibit deconsolidative burning. The HMX region above the line at high pressure represents the range of deconsolidative burning seen for low-binder HMX explosives (5-10 wt% binder, e.g., LX-07, LX-10, PBX-9501). In contrast to HMX and Explosive D, LLM-105 and TATB do not exhibit deconsolidative burning even at high pressures. Note that the LLM-105 line represents results from a series of formulations with 6-7.5 wt% inert binder (Viton A or Kel-F 800), and the TATB line represents results with 5-7.5 wt% inert binder (Kel-F 800).
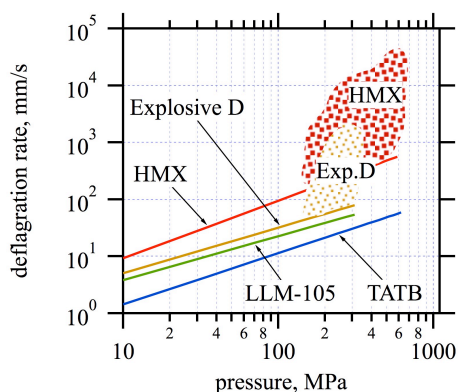

Fig. 9. Comparison of deflagration behavior for four explosives. Filled in areas at top right represent deconsolidative burning for Explosive D and for high-HMX explosives.

A couple of key points may be drawn from Figure 9. First, comparison across explosives for pressure regimes with no deconsolidative burning shows the decreasing rates in the sequence HMX / Explosive D / LLM-105 / TATB. These results are also included in Figure 4. Second, the propensity of the material to undergo deconsolidative burning either in its pure form (Explosive D) or in some formulations (low-binder HMX) indicates the likelihood for significantly higher rates of deflagration and hence energy release with these materials in situations where pressures may build up (statically or dynamically).

Unlike the properties and behaviors discussed previously, deflagration behavior does not necessarily indicate the ease with which a reaction can be initiated. It does directly indicate the expected reaction violence following initiation. That said, the deflagration behavior is certainly affected by some of the same parameters that govern reaction initiation. In solid samples (e.g., not deconsolidated), the burn front is propagated by heat from the flame being conducted into unburned explosive and causing it to decompose, generate gas, and then ignite. A material with a high ignition temperature, such as TATB, requires more heat for ignition, and therefore more time for heat flow and a slower deflagration rate.

In addition, grain scale features in the explosive may influence the deflagration properties. For example, different grain structures within a pure explosive such as Explosive D may lead to different likelihood for material fracture and deconsolidation, and therefore to different reaction violence for the same material.

Material Damage – Physical, Mechanical, Thermal

The effect of material damage on its sensitivity to ignition or reaction propagation may be profound. Damage may arise from physical, mechanical, or thermal stimuli. A thorough discussion of material damage and its effects are beyond the scope of this work, as there has been much work in this area, for example [2,14,15,18,19,20] and many others. I will present just a few examples.

An example of a physical stimuli is the imposition and subsequent release of external pressure. A sample of LX-04 pressure cycled to 200 MPa in the high pressure strand burner showed a very high and unexpected deflagration

rate, at the upper extreme of those shown in Figure 9. The cause was clear upon inspection of samples that were pressure cycled. As seen in Figure 10, the robust structure of this explosive with 15 wt% binder was completely disrupted after being pressure cycled. This was attributed to high-pressure argon diffusing into the Viton-A binder during pressurization; when the sample was rapidly depressurized the gas formed bubbles in the binder before it could diffuse out and physically disrupted the sample. This physical stimuli was not one that was anticipated to lead to increased reactivity, and this mechanism could be present in real systems with confinement that fails after buildup of gases through thermal decomposition.
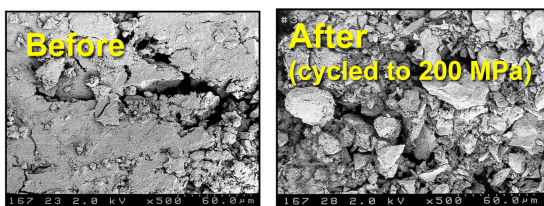

Fig. 10. LX-04 (85 wt% HMX, 15 wt% Viton A), before and after pressure- cycling with argon. 500X magnification.

A different form of damage may be incurred in material processing. For example, the sensitivity of Explosive D to shock is greatly increased after it has been pressed into and then removed from projectiles or other munitions.[21] While this type of processing should not change the inherent properties of the explosive, it almost certainly results in mechanical damage of the solid explosive.

Exposure of explosives to high temperatures may lead to chemical degradation or to physical changes that lead to sensitization. An example of this is heating of HMX explosives above the beta-to-delta phase transition. We have observed increases in the deflagration rate of LX-04, which does not otherwise exhibit deconsolidative burning, when heated above 433 K (the phase transition temperature);[14,15,23] the deflagration rates were similar to the deconsolidative burning rates in Figure 9. By pressurizing samples before heating, we were able to prevent the phase transition, and those samples had a much lower deflagration rate, thus demonstrating the importance of phase change on deflagration and reaction propagation.

Other effects of thermal damage include increasing increased permeability and deflagration rate for materials that do not undergo phase change.[19] Such changes can lead to the onset of deconsolidative burning and increased reaction violence.

Manifestation of Relevant Material Properties in Explosive Sensitivity

Consideration of the material properties discussed above leads to an expected ordering of explosive sensitivity among the four materials under discussion. For all properties, HMX is the most sensitive and TATB is the least sensitive, and this comparison doesn't tell us much. Comparison of LLM-105 and Explosive D is more fruitful. Chemical structure considerations favor LLM-105 stability – presence of an aromatic ring and nitro and amino groups. The higher thermal conductivity of LLM-105 compared with Explosive D allows hot spot energy to dissipate more rapidly and therefore requires a greater energy input for otherwise equal conditions. LLM-105 also has more favorable thermal stability – higher DSC onset temperature – while both LLM-105 and Explosive D show increase in onset temperature with increasing pressure. ODTX data are similar for both at high temperature, and the longer time to explosion at low temperatures with Explosive D presumably results from its lower thermal conductivity. Finally, for a given pressure the deflagration rate for LLM-105 is lower than that of Explosive D, and LLM-105 does not exhibit deconsolidative burning while Explosive D does at high pressures. Based on these properties, it would be reasonable to expect LLM-105 to be more less sensitive than Explosive D.

Figure 11 compares the sensitivity of these four explosives to mechanical impact (drop hammer impact) and to shock (gap test). The results are consistent with the above summary of material properties – HMX and TATB define the lower and upper bounds of sensitivity, with Explosive D being somewhat more sensitive than LLM-105.
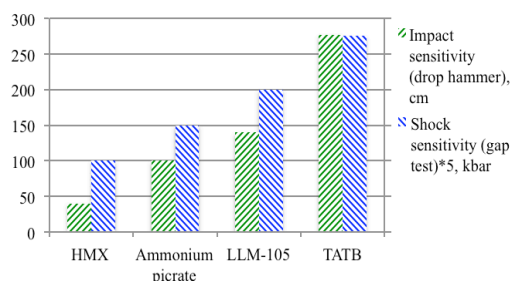
Fig. 11. Impact and shock sensitivity comparison (note shock sensitivity data have been multiplied by 5 to fit to scale).

A similar comparison may be drawn for the violence of thermal explosion as measured in the Scaled Thermal Explosion Experiment.[17,22] In this test a cylindrical explosive sample (~ 50 mm diameter, ~ 200 mm length) is confined in a sealed steel tube and heated until explosion. The violence is assessed by measurement of the wall velocity. More details are given elsewhere.[17,22] HMX-based explosives have been tested under many configurations (binder and binder wt%, confinement strength, heating rate), and exhibit wall velocities ranging from very little reaction to essentially full detonation.[22] In the only test with Explosive D, the violence was fairly low and there was considerable unreacted material expelled from the vessel during the test.[17] One STEX run was executed with PBX 9502, and was so benign that the vessel leaked the built-up gases and there was no explosion (if the vessel had not leaked, it would have eventually burst from the building pressure). There are no STEX data with LLM-105. In the absence of LLM-105 data and with the limited data for Explosive D, the STEX results are in agreement with the expected order of sensitivity as discussed above, consistent with analysis of the material properties.

Prediction of Explosive Insensitivity Based on Inherent Material Properties

The material properties and the resulting material behaviors presented and discussed above form at least a partial basis for determining the sensitive or insensitive nature of explosives. The presence of an aromatic ring, nitro and amino groups in a structure that promotes hydrogen bonding, absence of C-NO$_2$ bonds, and a crystal morphology that allows crystal shear without generation of high temperatures are all key molecular and crystallographic properties. High thermal conductivity and high thermal stability as shown by a high temperature for the DSC exotherm are also important; the pressure dependence of the DSC onset temperature may also help stabilize the explosive for materials with a positive slope in onset temperature vs. pressure. ODTX data provide insight into the thermal sensitivity, although these are the result of an integrated set of mechanisms and interpretation may be misleading (e.g., longer reaction times for Explosive D apparently don't indicate greater thermal stability but low thermal conductivity). Finally, the pressure-dependent deflagration rate and any occurrence of deconsolidative burning will have a significant effect on ultimate reaction violence. Just as these properties correctly predict the relative sensitivity of LLM-105 and Explosive D, they can be compared for other explosives to evaluate relative insensitivity. It is possible that, through integrated modeling of explosive response, threshold values for these parameters can be determined which determine if an explosives qualifies as an IHE.

Summary and Conclusions

By comparing several material properties and behaviors across a set of explosives ranging from fairly sensitive to insensitive, a consistent picture has been formed of the physical and chemical basis for the insensitive nature of IHEs. The relevant properties and behaviors are summarized in the paragraph immediately preceding this one. Currently TATB is the only explosive qualified as an IHE. By understanding the properties that underlie its unique nature, the prospects for development of new explosive molecules with improved performance while maintaining similar insensitivity may be enhanced.

Acknowledgments

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